

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- **BLACK BORDERS**
- **TEXT CUT OFF AT TOP, BOTTOM OR SIDES**
- **FADED TEXT**
- **ILLEGIBLE TEXT**
- **SKEWED/SLANTED IMAGES**
- **COLORED PHOTOS**
- **BLACK OR VERY BLACK AND WHITE DARK PHOTOS**
- **GRAY SCALE DOCUMENTS**

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**

PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C05G 5/00, C05B 19/00, C05C 3/00, 9/00, C05D 1/02, B01J 2/00		A1	(11) International Publication Number: WO 00/00452 (43) International Publication Date: 6 January 2000 (06.01.00)
(21) International Application Number: PCT/FI99/00568 (22) International Filing Date: 28 June 1999 (28.06.99) (30) Priority Data: 981490 29 June 1998 (29.06.98) FI 982013 18 September 1998 (18.09.98) FI		(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).	
(71) Applicant (<i>for all designated States except US</i>): KEMIRA AGRO OY [FI/FI]; Porkkalankatu 3, FIN-00180 Helsinki (FI). (72) Inventors; and (75) Inventors/Applicants (<i>for US only</i>): VAN BREMPT, Arthur [BE/BE]; Daalstraat 111, B-1852 Grimborgen (BE). POUKARI, Juhani [FI/FI]; Tinankuja 4 D 24, FIN-12430 Masala (FI). (74) Agent: BERGGREN OY AB; P.O. Box 16, FIN-00101 Helsinki (FI).		Published <i>With international search report.</i>	
(54) Title: A PROCESS FOR THE PREPARATION OF COMPOUND FERTILIZER GRANULES			
(57) Abstract <p>The invention relates to a process for the preparation of compound fertilizer granules containing at least two of the plant nutrients nitrogen, phosphorus and potassium, said process comprising the steps of: providing a solid feed material comprising at least one solid fertilizer raw material and optionally recycle material, feeding the feed material or a part thereof into a melter for melting a desired portion thereof and keeping said portion in molten state, feeding the molten or partly molten material and optionally other desired solid raw materials to a granulator to obtain a granulated product, and cooling and optionally screening the granulated product to obtain dry compound fertilizer granules having a desired size distribution, provided that no water or aqueous liquid is introduced into the process.</p>			

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		

A process for the preparation of compound fertilizer granules

The present invention relates to a process for the preparation of compound fertilizer granules by using solid granulation.

5 The term "compound fertilizer" is defined and used with several different meanings; it contains at least two of the plant nutrients nitrogen, phosphorus, and potassium. Compound fertilizers are produced chemically or by blending. They shall be in the form of granules, pellets, prills, or crystals and shall be free-flowing.

Compound fertilizers are manufactured and frequently used because they are 10 convenient to produce, transport, store and apply and because they fulfil local or regional nutrient requirements, especially basal nutrient needs well. In addition to containing various ratios of the primary nutrients ($N + P_2O_5 + K_2$), the compound fertilizers may contain certain secondary and micronutrients specific to the crop needs in particular agroclimatic regions.

15 Granulated fertilizers have several advantages over powders, particularly in decreasing the quantity of dust, improving uniformity of flow when fertilizers are applied and segregation when they are blended.

In classifying the granulation methods, the physical properties of the materials to be 20 granulated will be used. According to the density of the materials the following three groups may be distinguished: granulation of solids, granulation of slurries or of melts and granulation of fluids simultaneously with the reaction by which the product is formed.

Basic processes for manufacturing compound fertilizers are: Steam/water 25 granulation, Chemical granulation or complex or slurry granulation, Drop forming or prilling, Compaction granulation and Dry mixing or blending.

The principal mechanisms responsible for initial fertilizer granule formation and 30 subsequent growth are agglomeration and accretion. The known and widely applied compound fertilizer granulation methods are well reported for example in "Fertilizer Manual", Kluwer Academic Publishers, 1998, p. 434-451 and "Studies of Granulation of Compound Fertilizers Containing Urea: A Literature Review", G.C. Hicks, National Fertilizer Development Center; Bull Y-108, 15 pp., 1976.

Accretion is a process in which layer upon layer of a fluid material is applied to a solid particle causing it to grow in size; for example slurry-type granulation processes used to produce DAP, MAP, TSP, and some nitrophosphate compounds are accretion-type of granulation processes.

5 Agglomeration or granulation of solid particles is a classical method to granulate fertilizers, for example NPK products. In most agglomeration-type NPK formulations, 50-75% of the raw materials are fed as solid particles. The (premixed) raw materials are fed to a granulator where agglomeration is initiated. In the granulator steam and/or water or other liquid is added to provide sufficient liquid to
10 enhance granulation. In same processes a small amount ammonia may also be added to promote granulation and improve product quality by increasing the CHR (critical relative humidity) and decreasing the acidity. The solid particles are assembled and joint into granules by a combination of mechanical interlocking and cementing.

15 A number of industrial scale processes to manufacture compound fertilizers are developed and applied. In the steam/water granulation process steam and/or water or scrubber liquid is added into the granulator to provide sufficient liquid phase and plasticity to cause the dry raw materials to agglomerate into the product-size granules required.

20 The use of urea as a N-source for fertilizers of the different types and grades has established. Solid urea with quite high biuret content (0,8-2,0 wt-%) is mainly used for direct application to the soil and weak aqueous urea solutions with low biuret (max 0,3 wt-%) are used as foliage sprays.

The use of urea has also established in the manufacture of (granular) compound fertilizers based on for example superphosphate or ammonium phosphate.

25 The conventional wet granulation is not a suitable method for producing formulations containing urea, particularly when potassium chloride is also present, because the product is very hygroscopic and therefore difficult and expensive to dry.

30 In the chemical granulation beside a great amount of solid raw materials, water, steam, scrubber liquid, and/or ammonia and acid are fed to the granulator; the granules are formed mostly by agglomeration but in some processes granule formation may occur by accretion, too.

Drop formation or prilling, compaction granulation, dry mixing or blending, etc. are also applied quite widely to produce different granular fertilizer formulations.

Because some water or moisture is always present in most traditional granulation processes, drying is an obligatory, difficult and expensive stage of the processes and 5 cause the need of a separate dryer construction. To solve problems of granulation, product quality and drying different fertilizer grade-dependent processes are development.

A granulation process is described by Doshi, S.R. in the article "Fusion blend", Fertilizer Research vol. 30(1): p.87-97, 1991. Water (or steam) has been used to 10 agglomerate solids either in powder, prilled or granulated form but no other liquids such as ammonia, phosphoric acid, or nitric acid are involved in the described process; still drying is essential.

Some water or moisture is always included in the process. The process is temperature and material moisture-related. For example, for the most 15 agglomeration-type NPK fertilizers a liquid phase of about 300 kg/t of product is shown to be optimal.

Patent publication GB 1,189,398 (Sumitomo) discloses a process for producing a NK fertilizer which process comprises spraying a liquid mixture of urea, potassium chloride, gypsum and 1-10% by weight of water onto the solid material in a 20 granulator. No drying is used. However, the amount of water added into the process is high enough to keep urea in dissolved state and the final product has a quite great water content of 1-2% by weight.

Patent publication US 4,138,750 (TVA) discloses a process for the production of fertilizers from phosphoric acid, sulfuric acid, anhydrous ammonia and urea where a 25 specially designed pipe-cross reactor is used to produce a homogenous melt or slurry of low moisture content from phosphoric acid, sulfuric acid and anhydrous ammonia. The pipe-cross reactor eliminates the need for a preneutralizer and in addition, because of the low melt or slurry moisture content, the dryer is eliminated. The neutralization reaction heat is drying the material in the pipe-cross reactor.

30 Because of the water/moisture content of the raw materials and products, process and product quality problems, like increased hygroscopicity and plasticity, will often occur when fertilizers are granulated by using steam/water and chemical granulation processes; particularly when, for example SSP, TSP and/or urea is present in the product. The hygroscopicity and plasticity complicate drying,

screening and crushing operations, and furthermore, the storage properties of those compound fertilizers are often inferior to those of fertilizers that do not contain these substances.

The present invention is developed to solve the granulation, product quality, and storage etc. problems in manufacturing of compound fertilizers. The present invention relates to a process for the preparation of compound fertilizers such as NPK, NK etc., where solid raw materials are mixed in a mixer and fed to a granulator, whereto hot air is also fed. The raw materials are granulated without the aid of water or any other liquid such as ammonia, phosphoric acid or sulfuric acid.

Thus, the granulation is a true solid granulation process. Because water or any other liquid is not added, there is no need for drying the granulated product. Further more the physical quality of the product is good, too.

Particularly, the process of the present invention has great advantages to the known granulation methods which require higher temperatures at the drying phase.

Particularly, controlling of the humidity and drying temperature is important and difficult; the high temperature may cause melting of the granulated material and it will stick to the interior walls and flights of the dryer near the discharge end. The optimal values for humidity and temperature vary greatly from product to product.

Thus, the present invention provides a process for the preparation of compound fertilizer granules containing at least two of the plant nutrients nitrogen, phosphorus and potassium, said process comprising the steps of:

providing a solid feed material comprising at least one solid fertilizer raw material and optionally recycle material,

feeding the feed material or a part thereof into a melter for melting a desired portion thereof and keeping said portion in molten state,

feeding the molten or partly molten material and optionally other desired solid raw materials to a granulator to obtain a granulated product, and

cooling and optionally screening the granulated product to obtain dry compound fertilizer granules having a desired size distribution,

provided that no water or aqueous liquid is introduced into the process.

The melting of the feed material or a part thereof in the melter can be effected by introducing hot air into the melter. The melting can also be effected by other means, for example by heaters.

According to a preferred embodiment of the invention the process is carried out continuously, and the molten portion of the feed material is kept constant during the process by controlling the flow rate of the feed material and the temperature of the hot air introduced into the melter. The optimal proportion of the molten feed material is dependent on the grade of the fertilizer wanted and the raw materials used. The optimal proportion of the molten material can for example be about 10-40 wt-%, preferably about 10-25 wt-%, more preferably about 12-20 wt-%, depending on the grade.

When melting is carried out by the aid of hot air a suitable temperature of the hot air introduced into the melter is between 200 and 550 °C. At the melter outlet the hot air has a temperature of about 90 °C to 120 °C.

Suitably the temperature of the molten or partly molten feed material leaving the melter is between 70 °C and 135 °C, preferably between 70 °C and 110 °C.

The process of the invention can be carried out either by introducing all individual components of the raw materials into the melter or by introducing one or some of the individual components of the raw materials into the melter and the rest of the components into the granulator.

The material to be fed into the melter and/or granulator can be preheated. This is preferred in view of the temperature control of the process. The material can suitably be preheated to a temperature in the range from about 80 °C to about 110 °C.

The granulation temperature can vary depending on the formula of the fertilizer. The granulation temperature is preferably between about 75 °C and about 125 °C, more preferably between about 80 °C and about 125 °C.

The temperature of the cooled granulated product to be screened is typically between about 40 °C and 60 °C.

Typical solid fertilizer raw materials which can be used in the present invention are e.g. urea, diammonium phosphate (DAP), K₂SO₄ (SOP), monoammonium phosphate (MAP), phosphate rock, potassium chloride (MOP i.e. KCl), single superphosphate (SSP), triple superphosphate (TSP), ammonium sulfate (AS) and ammonium chloride (AC).

Preferably the fertilizer raw materials comprise urea, especially urea prills, and at least one other fertilizer raw material.

Additionally magnesium sulfate and/or one or several trace elements i.e. micro-nutrients, such as boron, can be added.

Furthermore bentonite, calcite, calcium oxide, calcium sulfate (anhydrous or hemihydrate), dolomite and/or sand and/or any other conventionally used filler can

5 be added.

According to the present invention all solid raw materials (solid fertilizer raw materials and optionally recycle material, micro nutrients and fillers) can be introduced into the melter. However, it is also possible to introduce a part of the solid raw materials into the melter and the remaining solid raw materials into the

10 granulator.

In a preferred embodiment the process of the invention comprise the step of screening the granulated product to obtain dry compound fertilizer granules having a size of 2 to 5 mm.

The undersize material (< 2 mm) and the oversize material (> 5 mm) obtained in the

15 screening can be recirculated as said recycle material. Optionally the oversize material can be milled after the screening before being recirculated. The temperature of the recycle material from the screening is typically about 60 °C or less.

The melter and granulator can be separate units but the melter and granulator can also be part of the same equipment.

20 This invention has advantages over the traditional granulation methods of the prior art technology because the raw materials are granulated without the aid of any water or any other liquid such as ammonia, phosphoric acid or sulfuric acid. Because water or any other liquid is not added, there is no need for drying of the product. This makes the granulation operation more simple and investment costs less
25 expensive because no separate equipment for drying is needed.

The final product will have a low water content (0.2-0.6 wt-%) originating from the raw materials. No supplementary drying is required. The water content of the products produced by traditional methods is normally about 1-2 wt-% causing already mentioned caking and applying problems.

30 The invention is illustrated in and by the following examples. Additionally the strength of the product granules obtained in the following examples was checked after a 3 months' storage, and the strength was found to be unchanged.

Example 1

Bench scale process for solid granulation.

Formulas (kg/t)

Raw material	GRADE		
	15-15-15 DAP+SSP	15-15-15 MAP+Sand	17-17-17 MAP+NH ₄ Cl
Urea	249	255	204
MAP(Lithuania 11-50)	-	300	340
DAP(Pernis 17-45)	210	-	-
SSP(Lithuania 19%)	287	-	-
NH ₄ Cl (N 26%)	-		153
KCl (K ₂ O 60%)	250	250	284
Sand	-	175	-

The mixture of the solid raw materials was fed to the bench scale granulator. Urea was added as prills. The melting of the mixture happened with hot air at the beginning of the granulator. Granulation was carried out at the granulator and partly at the cooler.

The process conditions and results are shown in Table 1.

Table 1

	GRADE		
	15-15-15 DAP+SSP	15-15-15 MAP+Sand	17-17-17 MAP+NH ₄ Cl

Process conditions:

Feed + recycle kg/h	8.3	10.1	11.9
Recycle ratio	0.2	0.2	0.2

Air heater

- temperature °C	336	316	322
- pressure bar	1.8	1.8	1.8

Temperature of product

granulator outlet °C	97	92	97
cooler outlet	30	32	35
Granulation	Good	Very good	Good

Product properties:

H ₂ O (KF)	%	0.25	0.15	0.28
N	%	15.2	16.1	18.2
P ₂ O ₅ total	%	15.9	15.0	17.1
K ₂ O	%	15.8	16.7	18.5
Granule strength N		52	40	50
Abrasion %		0	0.2	0.7
Shattering %		37	32	45
CRH %		34	35	43
Moisture absorption 80% RH				
2 h	%	2.8	2.7	3.2
4 h	%	5.7	5.5	6.2
6 h	%	8.8	8.3	9.1

The grade 15-15-15 granulated better when it contained MAP + sand than DAP + SSP.

The grade 17-17-17 containing ammonium chloride granulated good, too.

5 Ammonium chloride reacted partly with urea and formed urea · NH₄Cl. Nutrient content of each product was good. Physical properties of the products were good; the products were very dry.

Example 2

Bench scale process for solid granulation

	GRADE	NK 16-0-31	
		2A	2B
5	Formula	16-0-31	16-0-31
	Filler	Filler CaSO ₄	
	bentonite	hemihydrate	
	kg/t	kg/t	
	Urea (prills)	348	348
10	KCl (white)	517	517
	Bentonite	125	-
	CaSO ₄ *0.5H ₂ O (as dry matter)	-	125

15 The mixture of solid raw materials was fed with the recycle to the bench scale granulator. Melting happened with hot air at the beginning of the granulator. Granulation has been carried out at the granulator and partly at the cooler.

The products were coated with Esso coating oil 2 kg/t + talc 3 kg/t.

20 Very good or good granulation was obtained with a good product quality. However, the great humidity of air during the process caused some immediate increase of the water content of the final product.

The process conditions and results of the product tests are shown in Table 2.

Table 2**GRADE**

			NK 16-0-31	
			2A	2B
			16-0-31	16-0-31
5	Filler		Filler CaSO_4	
	bentonite		hemihydrate	
	Feed + recycle kg/h		9.0	9.0
	Recycle ratio		0.7	0.4
	Air heater			
10	- temperature	°C	294	238
	- pressure	bar	1.6	1.6
	Temperature of fert	°C		
	- granulator outlet		104	88
	- cooler outlet		28	27
15	Granulation		Very good	Good
	Product properties			
	Chemical analyses			
	Water (KF)	%	0.77	0.78
	Urea - N	%	16.6	16.8
20	N	%	16.6	16.8
	K ₂ O	%	31.8	30.9
	S	%	0.51	3.0
	pH		7.3	5.6
	Physical properties			
25	Granule strength	N	27	41
	Abrasion	%	1.3	1.1
	Volume weight	kg/l	0.77	0.80
	Flowability	kg/min	4.83	4.80
	Shattering	%	52	45
30	CRH	%	40	38
	Moisture absorption			
	80% RH			
	2 h	%	2.9	2.7
	4 h	%	5.0	4.5
35	6 h	%	7.0	6.8

Example 3

Bench scale process for solid granulation

GRADE	18-12-6+1.5MgO
5	kg/t
Urea (prills)	172
KCl (white)	100
Kovdor phosphate	155
DAP (Pernis) 17-45	143
10 AS (Leuna)	366
MgSO ₄	53

The mixture of solid raw materials was fed with the recycle to the bench scale granulator. Melting happened with hot air at the beginning of the granulator. Granulation has been carried out at the granulator and partly at the cooler.

15 The products were coated with Esso coating oil 2 kg/t + talc 3 kg/t.

Very good granulation was obtained with a good product quality. The process conditions and results of product tests are shown in Table 3.

Table 3

GRADE		
18-12-6+1.5MgO		
	Feed + recycle kg/h	9.0
	Recycle ratio	0.6
5	Air heater	
	- temperature °C	233
	- pressure bar	1.6
	Temperature of fert °C	
	- granulator outlet	98
10	- cooler outlet	28
	Granulation	Good
	Product properties	
	Chemical analyses	
	Water (KF) %	0.36
15	Urea - N %	8.5
	NH ₄ - N %	9.7
	N %	18.2
	P ₂ O ₅ - Total %	11.3
	P ₂ O ₅ - NAC %	6.0 (53%)
20	P ₂ O ₅ - WS %	5.5 (49%)
	K ₂ O %	8.4
	Mg %	1.3
	S %	10.8
	pH	5.8
25	Physical properties	
	Granule strength N	41
	Abrasion %	0.6
	Volume weight kg/l	0.84
	Flowability kg/min	4.88
30	Shattering %	59
	CRH %	40
	Moisture absorption	
	80% RH	
	2 h %	3.3
35	4 h %	5.2
	6 h	

Example 4

Bench scale process for solid granulation

5	GRADE	12-12-17+2 MgO + 0.5 B₂O₃
		kg/t
	Urea (crushed)	264
	Morocco phosphate	270
	TSP (P ₂ O ₅ 45%)	89
10	KCl (white)	284
	MgSO ₄	64
	Colemanite	6

15 The mixture of solid raw materials and recycle was preheated to about 100 °C in the feeding screw of the granulator. Melting happened with hot air at the granulation drum. Granulation has been carried out at the granulator and partly at the cooling drum.

The products were coated with SK Fert FW5 AG 2 kg/t + talc 3 kg/t.

Very good or good granulation was obtained with a good product quality. The process conditions and results of product tests are shown in Table 4.

Table 4

GRADE		12-12-17+2 MgO + 0.5MgO
Feed + recycle	kg/h	5.3
Recycle ratio		0.6
5	Granulation temperature °C	About 120
Air from cooler	"	27
Granulation		Good
Product properties		
Chemical analyses		
10	Water (Kf) %	0.35
	Urea - N %	12.4
	P ₂ O ₅ - Total %	12.2
	P ₂ O ₅ - NAC %	6.0 (49%)
	P ₂ O ₅ - WS %	2.8 (23%)
15	K ₂ O %	18.8
	Mg %	1.5
	B %	750
	pH	4.8
Physical properties		
20	Granule strength N	40
	Abrasion %	0.1
	Volume weight kg/l	0.82
	Flowability kg/min	5.4
	CRH %	23
25	Moisture absorption	
	80% RH	
	2 h %	3.2
	4 h %	5.5
	6 h	8.0

Example 5**Bench scale process for solid granulation**

5	GRADE	12-6-24
		kg/t
	Urea (crushed)	264
	SSP (P ₂ O ₅ 20%)	100
	Morocco phosphate	130
10	KCl (white)	400
	Colemanite	6
	Bentonite	80

15 The mixture of solid raw materials and recycle was preheated to about 100 °C in the feeding screw of the granulator. Melting happened with hot air at the granulation drum. Granulation has been carried out at the granulator and partly at the cooling drum.

The products were coated with SK Fert FW5 AG 2 kg/t + talc 3 kg/t.

20 Very good or good granulation was obtained with a good product quality. The process conditions and results of product tests are shown in Table 5.

Table 5

GRADE		
12-6-24		
	Feed + recycle kg/h	5.1
	Recycle ratio	0.84
5	Granulation temperature °C	About 120
	Air from cooler "	28
	Granulation	Very good
Product properties		
Chemical analyses		
10	Water (KF) %	0.27
	Urea - N %	13.1
	P ₂ O ₅ - Total %	6.0
	P ₂ O ₅ - NAC %	2.9 (48%)
	P ₂ O ₅ - WS %	0.84 (14%)
15	K ₂ O %	25.8
	B %	850
	pH	6.1
Physical properties		
	Granule strength N	39
20	Abrasion %	0.1
	Volume weight kg/l	0.84
	Flowability kg/min	5.6
	CRH %	15
Moisture absorption		
25	80% RH	
	2 h %	2.1
	4 h %	4.1
	6 h	6.0

Example 6

GRADE	
15-15-15	
5	
Urea (crushed)	285 kg/t
Urea melted	100 %
DAP	117 kg/t
Yunnan rock phosphate	330 kg/t
10 MOP	255 kg/t
Bentonite	6 kg/t

15 Urea was melted in a separate reactor and mixed with the other raw materials preheated to 90 °C. The temperature at the beginning of the granulation was 110.4 °C and at the end of the granulation 103.2 °C. The duration of the granulation stage was 4 minutes.

Product properties:

H ₂ O (KF)	0.09
20 Granule strength N	34.5

Very good granulation was obtained.

Example 7

25 Bench scale process for solid granulation

GRADE	
15-15-15	
30	
Urea (46 %)	276 kg/t
DAP (17-45)	142 kg/t
Rock phosphate (P ₂ O ₅ 32 %)	270 kg/t
35 K ₂ SO ₄ (K ₂ O 50 %)	300 kg/t

The mixture of solid raw materials and recycle was preheated to about 100 °C with IR in the feeding screw of the drum. The outer wall of the granulation drum was

heated with IR too. Urea was crushed beforehand. Small amount of hot air was used in the melting of urea in the granulation drum. Drying drum worked as cooler.

The products were coated with SK Fert FW5 AG 2 kg/t + talc 3 kg/t.

5

Process conditions:

Feed and recycle kg/h	5.07
Recycle ratio	0.75
Recycle heater °C	179

10

Granulation drum

x Outside	°C	268
x Inside	"	117
Air to the drum	"	287
Air to cooler	"	24
Air from cooler	"	28
Granulation		Good

Product properties:

H ₂ O (KF)	%	0.09
N	"	15.5
P ₂ O ₅ total	"	15.4
K ₂ O	"	16.1
S	"	6.6

25

Granule strength N	30
Abrasion %	0.4
Shattering %	28
CRH %	18

30

Moisture abs.	
80 % RH	
2 h %	2.6
4 h %	4.8
6 h %	6.6

35

SOP based 15-15-15 granulated good.

Claims

1. A process for the preparation of compound fertilizer granules containing at least two of the plant nutrients nitrogen, phosphorus and potassium, said process comprising the steps of:
 - 5 providing a solid feed material comprising at least one solid fertilizer raw material and optionally recycle material, feeding the feed material or a part thereof into a melter for melting a desired portion thereof and keeping said portion in molten state, feeding the molten or partly molten material and optionally other desired solid raw materials to a granulator to obtain a granulated product, and cooling and optionally screening the granulated product to obtain dry compound fertilizer granules having a desired size distribution, provided that no water or aqueous liquid is introduced into the process.
 - 10
 2. A process according to claim 1, wherein the process is carried out continuously, and the molten portion of the feed material is kept constant during the process by controlling the flow rate of the feed material and the temperature of the melter.
 - 15
 3. A process according to claim 1 or 2, wherein the temperature of the molten or partly molten feed material is between 70 °C and 135 °C.
 - 20
 4. A process according to any of claims 1-3, wherein the melting is effected by introducing hot air into said melter.
 5. A process according to claim 4, wherein the temperature of the hot air introduced into the melter is between 200 °C and 550 °C.
 - 25
 6. A process according to any of claims 1-5, wherein from 10 to 40% by weight of the feed material melts in the melter.
 7. A process according to any of claims 1-6, wherein said solid feed material to be fed into the melter comprises all individual components of the raw materials.
 - 30
 8. A process according to any of claims 1-6, wherein said solid feed material to be fed into the melter comprises one or some of the individual components of the raw materials, and the rest of the components is fed to the granulator.

9. A process according to any of claims 1-8, wherein said solid feed material to be fed into the melter is preheated.
10. A process according to any of claims 1-9, wherein the solid raw material to be fed to the granulator is preheated.
11. A process according to claim 9 or 10, wherein the material is preheated to a temperature in the range from 80 °C to 110 °C.
- 10 12. A process according to any of claims 1-11, wherein the granulation temperature is in the range from 75 °C to 125 °C, preferably from 80 °C to 125 °C.
13. A process according to any of claims 1-12, wherein the fertilizer raw materials are selected from the group consisting of urea, diammonium phosphate (DAP),
15 K_2SO_4 (SOP), monoammonium phosphate (MAP), potassium chloride (MOP), phosphate rock, single superphosphate (SSP), triple superphosphate (TSP), ammonium sulfate (AS) and ammonium chloride (AC).
14. A process according to claim 13, wherein the fertilizer raw materials comprise urea and at least one other of said fertilizer raw materials.
- 20 15. A process according to any of claims 1-14, wherein additionally at least one material selected from the group consisting of magnesium sulfate and micronutrients is introduced into the process.
16. A process according to any of claims 1-15, wherein additionally at least one
25 filler selected from the group consisting of bentonite, calcite, calcium oxide, anhydrous calcium sulfate, calcium sulfate hemihydrate, dolomite, and sand, is introduced into the process.
17. A process according to claim 1, wherein the undersize material and the oversize material obtained in the screening are recirculated as said recycle material, said oversize material optionally being milled after the screening.
- 30 18. A process according to any of claims 1-17, wherein the moisture content of the dry compound fertilizer granules is below 0.6% by weight, preferably below 0.3% by weight.

INTERNATIONAL SEARCH REPORT

International application No..

PCT/FI 99/00568

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: C05G 5/00, C05B 19/00, C05C 3/00, C05C 9/00, C05D 1/02, B01J 2/00
 According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: C05B, C05C, C05D, C05G, B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPIL, EDOC, JAPIO

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4008064 A (ÖYVIND SKAULI), 15 February 1977 (15.02.77), column 1, line 1 - line 12; column 2, line 13 - column 3, line 35; column 4, line 27 - column 8, line 22, figure 1, claims 1, 4-6, abstract --	1-18
X	GB 1462633 B (FISONS LIMITED), 26 January 1977 (26.01.77), page 1, line 21 - page 2, line 82, claim 4 --	1-18

 Further documents are listed in the continuation of Box C. See patent family annex.

* Special categories of cited documents:	
"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E" earlier document but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search	Date of mailing of the international search report
4 October 1999	18-10-1999
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. + 46 8 666 02 86	Authorized officer Ingrid Grundfält/MP Telephone No. + 46 8 782 25 00

INTERNATIONAL SEARCH REPORT

International application No..

PCT/FI 99/00568

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 55015655 A (HITACHI METALS LTD) 1980-02-02 (abstract) World Patents Index (online). London U.K.: Derwent Publications, Ltd. (retrieved on 1999-10-04). Retrieved from: EPO WPI Database. DW 8011, Accession No 80-19312C & JP 55015655 (HITACHI METALS LTD) 1980-02-02 (abstract) (online) (retrieved on 1999-10-04) Retrieved from: EPO PAJ Database. --	4
A	US 4398936 A (JOHAN W. HOGENDONK ET AL), 16 August 1983 (16.08.83), column 1, line 33 - line 47; column 1, line 66 - column 2, line 5; column 2, line 39 - column 3, line 62, column 4, line 34 - line 40 --	1,8,10,13
A	JP 60065785 A DAIYA CHEMICAL KK et al 1985-04-15 (abstract) World Patents Index (online). London, U.K.: Derwent Publications, Ltd. (retrieved on 1999-10-04). Retrieved from: EPO WPI Database. DW 198521. Accession No. 1985-126615. --	1,15
A	US 5676729 A (JIM L. ELROD ET AL), 14 October 1997 (14.10.97), column 2, line 16 - line 21; column 3, line 15 - column 4, line 27, claim 1 --	1,13,16
A	US 4410350 A (GEORGE G. JUDD), 18 October 1983 (18.10.83), column 1, line 11 - line 36; column 2, line 66 - column 3, line 15, claim 1, abstract --	1,13,14,16
A	WO 9629287 A1 (DEAD SEA WORKS LTD.), 26 Sept 1996 (26.09.96), page 2, line 11 - page 3, line 12; page 4, line 22 - page 8, line 18, figures 1,2 --	1-18

INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 99/00568

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 1159445 B (PRODUITS CHIMIQUES PECHINEY-SAINT-GOBAIN), 23 July 1969 (23.07.69), page 1, line 43 - page 2, line 44; page 2, line 109 - page 4, line 26, claims 1-7,14,15 -----	1-18

INTERNATIONAL SEARCH REPORT

Information on patent family members

30/08/99

International application No.

PCT/FI 99/00568

Patent document cited in search report	Publication date	Patent family member(s)	Publication date	
US 4008064 A	15/02/77	AU 8335975 A BE 831779 A BG 39806 A BR 7504741 A CA 1063778 A DD 118995 A DE 2533328 A,C DK 137068 B,C DK 338275 A EG 11720 A FI 62628 B,C FI 752127 A FR 2279457 A,B GB 1490003 A IE 41302 B IN 142375 A IT 1040154 B JP 1115790 C JP 51037098 A JP 56048202 B KE 2852 A NL 184097 B,C NL 7508914 A OA 5060 A RO 71993 A SE 395617 B,C SE 7508183 A TR 18469 A YU 40444 B YU 188775 A	27/01/77 17/11/75 15/08/86 06/07/76 09/10/79 05/04/76 12/02/76 16/01/78 27/01/76 29/03/78 29/10/82 27/01/76 20/02/76 26/10/77 05/12/79 02/07/77 20/12/79 29/09/82 29/03/76 14/11/81 04/08/78 16/11/88 28/01/76 31/12/80 30/06/81 22/08/77 28/01/76 24/02/77 28/02/86 28/02/82	27/01/77 17/11/75 15/08/86 06/07/76 09/10/79 05/04/76 12/02/76 16/01/78 27/01/76 29/03/78 29/10/82 27/01/76 20/02/76 26/10/77 05/12/79 02/07/77 20/12/79 29/09/82 29/03/76 14/11/81 04/08/78 16/11/88 28/01/76 31/12/80 30/06/81 22/08/77 28/01/76 24/02/77 28/02/86 28/02/82
GB 1462633 B	26/01/77	NONE		
US 4398936 A	16/08/83	AT 535 T BR 7904199 A CA 1120282 A EG 14816 A EP 0007132 A,B SE 0007132 T3 GR 72920 A IE 48161 B IN 152174 A JP 55010494 A JP 61009277 B NL 7807219 A SU 1056896 A TR 20420 A YU 40566 B YU 147679 A	15/01/82 18/03/80 23/03/82 30/06/85 23/01/80 08/01/80 23/11/83 26/06/81 28/02/86 21/01/83	
US 5676729 A	14/10/97	AU 697843 B AU 6184296 A EP 0835232 A WO 9701518 A	15/10/98 30/01/97 15/04/98 16/01/97	
US 4410350 A	18/10/83	NONE		

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.-

PCT/FI 99/00568

30/08/99

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
WO 9629287 A1	26/09/96	AU 5146696 A		08/10/96
		CA 2213738 A		26/09/96
		IL 113104 A		26/01/99
GB 1159445 B	23/07/69	BE 699808 A		12/12/67
		DE 1592734 A		10/02/72
		DK 130632 B,C		17/03/75
		ES 341700 A		01/07/68
		NL 6708178 A		14/12/67